Dietary Fiber Methods in Codex Alimentarius: Current Status and Ongoing Discussions

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Background

The global market for the international food trade is ever-increasing, and a uniform set of standards and procedures is critical to ensuring foods are safe and trade practices are fair (1).

The Codex Alimentarius Commission (CAC) develops harmonized international food standards, guidelines, and codes of practice to protect the health of consumers and ensure fair practices in the food trade. Established in 1963 by FAO (Food and Agriculture Organization of the United Nations) and WHO (World Health Organization), the Codex Alimentarius (or “food code” when translated to English) has become the global reference point for food producers, processors, and regulators, as well as a potential resource for consumers to aid their understanding of the international food safety and trade system. Membership in CAC is open to all member nations and associate members of FAO and WHO. Non-governmental organizations, like AACC International (AACC), do not have direct voting rights, as members do, but can participate in the work of CAC when granted “observer status.” This status allows AACC to send representatives to CAC and its related meetings to provide scientific guidance and express the views and opinions of AACC and its members.

Within the CAC structure, a number of subsidiary bodies, including general subject committees and commodity committees, help prepare draft standards for submission to the commission. The general subject committees can be thought of as “horizontal committees” because their work is relevant for all the commodity committees and standards. The most relevant committees with respect to dietary fiber methods of analysis are the Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU) and the Committee on Methods of Analysis and Sampling (CCMAS). CCNFSDU develops general provisions concerning the nutritional aspects of all foods, including special diets, and develops standards and guidelines for these foods. CCMAS ensures that appropriate analytical methods are specified to support the guidance documents and standards put forth by CAC.

There are principles for the establishment of Codex Methods of Analysis, and these are published in the Codex Procedural Manual (2). The methods specified are intended primarily for the verification of provisions in Codex standards and can be used as reference methods or for routine and control purposes. The principles cover a method classification system that describes different types of methods of analysis. There are four different types of Codex methods described in the manual; “Defining Methods (Type I),” “Reference Methods (Type II),” “Alternative Approved Methods (Type III),” and “Tentative Methods (Type IV).” This “typing” can be clarified further by relating what each method measures or quantifies. Type I methods are referred to as “defining/empirical” or non-analyte-specific methods, i.e., the quantity measured is defined by the result found by following the stated method. Type II and III methods are referred to as “rational” methods, in which an identifiable chemical or analyte is quantitated; Type II methods are used for resolving disputes regarding analyte level. Type IV methods are considered “tentative” methods that can be either empirical or rational but that have not met all criteria required for acceptance.

Dietary Fiber Definition and the Need for Related Methods

The current accepted definition for dietary fiber in the “Guidelines on Nutrition Labeling” (3) resulted from a 16 year discussion within CCNFSDU and was recommended to, and accepted by, CAC at the July 2009 meeting (4). Shortly thereafter, AACC Approved Method 32-45.01, which quantitates dietary fiber per the accepted Codex definition, was developed. The method compendium “Codex Standard 234,” Recommended Methods of Analysis and Sampling, was updated in 2011 to include this method following the decision of CCMAS. In 2011, AACC Approved Method 32-50.01, which quantitates dietary fiber and its subcomponents, insoluble and soluble dietary fiber (IDF and SDF, respectively), per the Codex definition, was developed and is awaiting consideration by CCNFSDU and CCMAS.

The current “Codex Standard 234” method compendium contains the table "Methods of Analysis for Dietary Fibre: Guidelines for Use of Nutrition and Health Claims: Table of Conditions for Claims,” which lists eight Type I methods, five Type II methods, one Type III method, and three Type IV methods for dietary fiber analysis or components thereof. Methods that report a result as “dietary fiber” are considered Type I as they are non-analyte specific and are empirical. The most widely used of these methods are AACC Approved Method 32-05.01 (AOAC 985.29), AACC Approved Method 32-07.01 (AOAC 991.43), and AACC Approved Method 32-45.01 (AOAC 2009.01). Having eight, and possibly nine when AACC Approved Method 32-50.01 (AOAC 2011.25) is considered, Type I methods creates additional confusion because there should be only a single Type I method (per the Codex principles) that can be used to determine the accepted value of the “item measured.” In this case, the item measured is dietary fiber, as determined by a particular defining method. The current table does include additional method information, such as principle, reference, and provisions for each method, but it does not specify the method to use in a specific situation or with a specific product type.
Fig. 1. Decision tree proposed in discussion paper on selection of methods of analysis for dietary fiber.
Those familiar with dietary fiber testing understand the complexity of the analyses involved and see value in the different testing approaches one could use to obtain a result for dietary fiber in different food matrices. This may explain why there are multiple dietary fiber methods listed as Type I, since results will differ depending on the ingredients and fiber components in the sample being tested. Those unfamiliar with dietary fiber testing may view the multiplicity of Type I methods as a further complication to selecting the most appropriate method of analysis. This view was noted at the thirty-second session of CCMAS and led to the establishment of an electronic working group that would consider the elaboration of a decision tree to aid an analyst’s selection of the most appropriate method of dietary fiber analysis.

CCMAS Session Thirty-Three

Prior to the thirty-third CCMAS meeting (CCMAS 33), the dietary fiber electronic working group, which included AACCI, generated a discussion paper on the use of a decision tree for selecting the appropriate method of analysis for dietary fiber (5). Part of the discussion involved the selection of methods for dietary fiber analysis and potential tools to help analysts determine the most appropriate method to use. It was noted by the working group that a number of member delegations had supported the development of a decision tree at the thirty-second session, while others did not.

Figure 1 is an example of a decision tree from the discussion paper prepared by the electronic working group (5). The objective of this decision tree is to help analysts in making the correct decision regarding dietary fiber methodology. This may seem beneficial to someone who is inexperienced with dietary fiber chemistry, but one limitation of this tree is that in order to accurately follow the decision process, one needs to have a thorough understanding of dietary fiber testing methods. An analyst with this level of understanding would more than likely already know which method would be most appropriate for the sample type being tested. The decision tree also incorporates aspects of the definition of dietary fiber that should be left to national authorities, such as consideration of “physiological benefit” and inclusion of “carbohydrates from 3 to 9 monomeric units,” factors that are outside the decision of which method to choose per se. Also included in the decision tree are methods that have not been recommended for use by CCMAS and, hence, are not specified in Codex Standard 234. Any information or guidance generated by CCMAS should only include methods that have been reviewed and adopted into the Codex standards. Step 3 of the tree includes a step to identify the amount of any added components present by detailed chemical analysis. Depending on the product type and dietary fiber ingredients, it may not be scientifically possible for the laboratory, nor economically feasible, to determine the chemical components, much less distinguish inherent versus added components, in a sample. In discussions on this option, many experts in the field of dietary fiber analytical chemistry felt that this decision tree (and possibly this approach) would further confuse analysts trying to determine which method to use and how to interpret the results.

Additional suggestions for guidance included the use of diagrams, tables, footnotes, and/or explanatory text in the standard itself. The diagrams in Figure 2, which are similar to those used in several previous publications, show the approximate amounts of certain dietary fiber components captured by the most widely performed methods. These diagrams show that some soluble dietary fiber components are not measured with gravimetric-only analyses and others are only partially measured. Analyzing products containing these partially measured components can create overestimation, because double counting can occur when using an analyte-specific method in conjunction with the gravimetric-only method. The most accurate approach would be to utilize a single method in which there is no possibility of double counting.

At the Working Group on Endorsement of Methods of Analysis meeting prior to CCMAS 33, it was generally accepted that dietary fiber is a very complex topic and there will ultimately be differences of opinion on the most appropriate testing approaches to meet the definition. It was also accepted that further clarity about methodology should be made available, but this should not be in the form of dictating a particular testing scheme but should only provide more information.

During the CCMAS 33 session it was ultimately decided that a table listing each dietary fiber method included in Codex Standard 234 should be prepared and that what is measured and what is not measured by each should be specified. This is available as conference room document (CRD) 16 from

![Fig. 2. Dietary fiber components measured by gravimetric-only methods (AACCI Approved Methods 32-05.01 and 32-07.01) and gravimetric-liquid chromatographic methods (AACCI Approved Methods 32-45.01 and 32-50.01).](image-url)
Ideal World of Dietary Fiber Analyses

In an ideal world, there would be a single method (AACCI 32-45.01 or AACCI 32-50.01 when measurement of IDF and SDF quantities are desired in addition to total dietary fiber [TDF] quantity) for the determination of dietary fiber per the Codex definition, and it would be the method of choice in all situations. There may be a number of factors that make this impossible, and one factor could be the definition itself. The definition states that the inclusion of carbohydrates with 3 to 9 monomeric units is left to national authorities, and currently, there is no method that quantitates dietary fiber with inclusion of all dietary fiber carbohydrate polymers greater than 10 monomers to the exclusion of all dietary fiber carbohydrate polymers with 9 monomers or fewer.

There are generally two approaches to take when testing a sample for dietary fiber: use a gravimetric-only method or use a gravimetric method plus further analysis of the normally disposed filtrate. There is a misconception that method AACCI 32-45.01 allows for the quantitation of only carbohydrates greater than 3 monomeric units; however, this method does allow for results to be obtained for only the gravimetric portion. Depending on the ingredients, what is captured could be very similar to what would be captured with a gravimetric-only method, as shown in Table I. Another misconception about method AACCI 32-45.01 is that it requires more time and instrument resources than traditional dietary fiber methods. This is true if the amount of water/alcohol soluble fiber (SDFS) needs to be determined, but if only the gravimetric determination is needed, then the resources needed to perform that portion of this method are similar to traditional dietary fiber methods.

Recently an additional analytical method for dietary fiber has been approved by AACCI and AOAC. AACCI 32-50.01 (AOAC 2011.25) captures the same types of material as AACCI 32-45.01 but includes an additional step to separate IDF from precipitated SDF. This allows the gravimetric portion of the analysis to be similar to that of the traditional method used for IDF and SDF determination, AACCI 32-07.01 (AOAC 991.43). It is again worth noting that Table I shows the similarities of materials captured with a traditional method (AACCI 32-05.01) and those obtained using the gravimetric portion of AACCI 32-45.01. The same similarities would also hold true for methods AACCI 32-07.01 and AACCI 32-50.01. If equivalent materials are captured using traditional methods and the gravimetric portion of newer methods, then the gravimetric portions of methods AACCI 32-45.01 and AACCI 32-50.01 could be used in place of methods AACCI 32-05.01 and AACCI 32-07.01.

Methods AACCI 32-45.01 and AACCI 32-50.01 are the most inclusive methods for determining dietary fiber and are ideal when testing a product that has unknown fiber ingredients. Even though these methods are currently the most comprehensive, they are not perfect and have been shown to underestimate some dietary fiber components. β-(2,6)-fructo-triose, which can be present in some types of commercial fructooligosaccharide preparations, is not captured because it coelutes with the disaccharide marker via chromatographic analysis. A solution to this problem has been developed and requires a minor modification to the approved method; this modification has been included in AACCI 32-50.01. Another concern that has been raised involves the analysis of certain types of chemically modified resistant starch(es). In the case of one RS4, it is partially digested during the initial enzymatic hydrolysis, leading to lower results when compared with traditional dietary fiber methods. In this situation, there is a debate as to which method provides the “correct” value for this ingredient. Regardless, the reality is that currently no single method is perfect for all situations and product types. However, AACCI 32-50.01 provides the most accurate estimate of dietary fiber, as defined by CAC, in foods and food products.

Summary

Currently there are multiple Type I methods for dietary fiber analysis, which conflicts with the principles of the Codex Procedural Manual (2). While not ideal, CCMAS is using the most recent scientific evidence to simplify and aid those involved in

<table>
<thead>
<tr>
<th>Method</th>
<th>What Is Measured</th>
<th>What Is Not Measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>AACCI 32-05.01/AOAC 985.29</td>
<td>• Insoluble dietary fiber</td>
<td>• Some resistant starches, dependent on type</td>
</tr>
<tr>
<td></td>
<td>• Some resistant starches (RS2, RS2, RS4), dependent on type</td>
<td>• Soluble dietary fiber that does not precipitate in 78% aqueous ethanol, including most inulin, polydextrose, and resistant maltodextrins</td>
</tr>
<tr>
<td>AACCI 32-45.01/AOAC 2009.01</td>
<td>• Insoluble dietary fiber</td>
<td>• Some RS4 resistant starches, dependent on type</td>
</tr>
<tr>
<td>Gravimetric determination only</td>
<td>• Resistant starches (RS2, RS2, RS4)</td>
<td>• Soluble dietary fiber that does not precipitate in 78% aqueous ethanol, including most inulin, polydextrose, and resistant maltodextrins</td>
</tr>
<tr>
<td></td>
<td>• Soluble dietary fiber that precipitates in 78% aqueous ethanol, including some inulin, polydextrose, and resistant maltodextrins</td>
<td>• Some RS4 resistant starches, dependent on type</td>
</tr>
<tr>
<td>AACCI 32-45.01/AOAC 2009.01</td>
<td>• Insoluble dietary fiber</td>
<td>• Some resistant starches, dependent on type</td>
</tr>
<tr>
<td>Gravimetric &amp; HPLC determination</td>
<td>• Resistant starches (RS2, RS2, RS4)</td>
<td>• Soluble dietary fiber that does not precipitate in 78% aqueous ethanol, including most inulin, polydextrose, and resistant maltodextrins and all FOS, AXOS, XOS, and GOS</td>
</tr>
<tr>
<td></td>
<td>• All soluble dietary fiber</td>
<td>• Some RS4 resistant starches, dependent on type</td>
</tr>
</tbody>
</table>
dietary fiber testing. At times, scientific understanding may outpace the rate at which information can be collected, reviewed, and disseminated, but the Codex Alimentarius is progressing using input from world experts in dietary fiber method research. Harmonizing an internationally accepted definition for dietary fiber was a large undertaking that spanned 16 years. Some national authorities have openly accepted this fairly recent definition, whereas others have not commented on the topic for a number of years. To ensure the most appropriate test method is used, analysts, laboratory managers, and others analyzing foods for dietary fiber need to understand how and where the results from the testing will be used and any legal implications that may arise. While CAC is working toward a single analytical method commensurate with its concise definition, there may not be a procedure that accounts for all possible situations. In the interim, CAC and its communities can aid analysts worldwide by providing guidance and information when possible. This report is intended to capture the concerns and issues raised by the electronic working group and during the meetings of CCMAS 33 and can be used to provide guidance and additional information to that supplied in a Codex standard.

Acknowledgments

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References